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⑭ 発明の名称 蛍光体およびその製造方法

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明 細 書

1. 発明の名称

蛍光体およびその製造方法

2. 特許請求の範囲

1. 一般式



$$(0 < a \leq 0.3, 0 < b \leq 0.1)$$

で表され、母体組成が硫化亜鉛と硫化マグネシウムの固溶系からなり、主発光が Tm^{3+} によりもたらされることを特徴とする蛍光体。

2. 特許請求の範囲第1項記載の蛍光体において、

$$(0.05 < a \leq 0.2, 0 < b \leq 0.06)$$

で表わされることを特徴とする蛍光体。

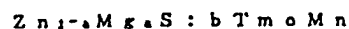
3. Tm 原料として、 Tm の硫化物を用い、所要量の Zn 、 Mg 原料と共に900℃以上で焼成することを特徴とする蛍光体の製造方法。

4. EL(エレクトロルミネセンス)素子を構成する発光層の少なくとも1成分が、特許請求の範囲第1項記載の蛍光体からなることを特徴とするEL素子。

5. Zn 、 Mg 、 Tm の各原料がすべて硫化物で構成され、それらを成膜することにより、特許請求の範囲第1項記載の組成を有する蛍光体膜を得るようにしたことを特徴とする蛍光体膜成膜用蒸発源ターゲット。

6. 特許請求の範囲第4項記載の発光層と、該発光層に対して補色関係が成立する橙色発光 $\text{ZnS} : \text{Mn}$ 発光層との組み合わせからなることを特徴とするEL素子。

7. 一般式



$$(0 < a \leq 0.3, 0 < b \leq 0.1, 0 \leq c \leq 0.1)$$

で表され、母体組成が硫化亜鉛と硫化マグネシウムの固溶系からなり、主発光が Tm^{3+} によりもたらされることを特徴とする蛍光体。

3. 発明の詳細な説明

〔産業上の利用分野〕

本発明は、主発光が青色領域にある新規組成の蛍光体ならびにその製造方法に係わり、特に白色EL(エレクトロルミネセンス)表示の青色成

分に用いて優れた特性を示す蛍光体に関する。

〔従来の技術〕

(Zn, Mg)Sを母体とする蛍光体の公知例として以下のような文献があるが、上記の母体にTm, Tm-Mnを付活した蛍光体についての公知例はない。

- 1) ジャーナル・オブ・エレクトロケミカル・ソサイエティ第99巻, 第4号, 第155頁から158頁[J. Electrochemical Soc. 99(4)155-158 (1952)]において、MgS 25モル%以下、残りZnSから成る(Zn, Mg)S母体にCu, Ag, Pb, As, Sb, BiおよびCu-Mn, Pb-Mn, Cu-Pbをそれぞれ付活した蛍光体について、紫外線励起ならびに電子線励起による発光特性が示されている。
- 2) ジャーナル・オブ・クリスタルグロース 第72巻, 第179頁から183頁[J. of Crystal Growth 72 179-183 (1985)]において、 $Zn_{1-a}Mg_aS$, $0 < a < 0.1$ の母体に50ppmのMnを付活すると、290nm励起により450

と報告されている。

- 6) 特開昭51-49397号には、赤色、緑色、青色のいずれか一色の発光EL素子を一枚の透明基板に形成し、残り二色の発光EL素子を別個の一枚の透明基板に形成し、両基板を対向して配置したことを特徴とするフルカラー薄膜EL素子が開示されている。

〔発明が解決しようとする課題〕

上記の公知例のうち、1), 2), 3)はいずれも主発光が青色領域にある蛍光体であるが、発光帯の半値幅が70nm以上に及びカラー表示に用いる青色成分としては彩度が低い。また、これらの蛍光体を出発原料に用いて作成した薄膜EL素子は全く発光しないか、発光しても極めて輝度が低い。

一方、公知例5)に示したZnS:TmF_aの青色発光色は彩度は高いが、 λ_m/λ_w はZnS:Mnのそれより2桁以上低いという欠点がある。

さらにまた、公知例6)は、素子構成が複雑になる欠点がある。

nmピークの発光が顕現されたと記載されている。

- 3) ジャーナル・オブ・マテリアルサイエンス第21巻, 第2100頁から2108頁[J. of Material Science 21 2100-2108 (1985)]において、 $Zn_{1-a}Mg_aS:Cu, Br$ を用いた分散型ELの発光ピーク波長は、 x の増大に伴い525nmから436nmまでシフトすると報告されている。
- 4) ラシアン・ジャーナル・オブ・インオーガニクケミストリー第9巻, 第4報, 第512頁から516頁[Russian J. of Inorganic Chemistry 9 (4)512-516 (1964)]においてMgSはZnSに対して22モル%まで固溶し、固溶量の増大に伴い六方晶系格子定数 a_0, c_0 がともに増大すると記載されている。
- 5) '80 エス・アイ・ディ ダイジェスト第106頁['80 SID Digest p106(1980)]には、薄膜EL素子においてZnS:TmF_aの λ_m/λ_w は、ZnS:Mnのそれより二桁小

本発明の目的は、上述の問題点を一挙に解決した新規の蛍光体組成物ならびにその製造方法を提供することにある。本発明の他の目的は、白色表示可能なEL素子を提供することにある。また、本発明のさらに他の目的は、上記EL素子発光体となる蛍光体膜の成膜に好適な蒸着源ターゲット組成物を提供することにある。

〔課題を解決するための手段〕

上記目的は、発光イオンに彩度の高いTm³⁺を用い、かつTm³⁺の導入をより容易ならしめるようにZnS系母体格子の拡火をはかることにより達成される。

すなわち、本発明の蛍光体の組成は一般式



で与えられ、 $0 < a < 0.3$, $0 < b \leq 0.1$

の範囲において最も効果的な特性が得られる。

ここで、上記組成に加えて、原料や製造工程において不可避免的に混入する不純物が存在することは、本発明の要旨を逸脱するものではない。

また、上記組成の蛍光体膜と、他の蛍光体膜と

原料は実施例1と同じTmF₃であり、蒸発源ターゲットは上記と同じ加圧成型焼結のプロセスを経て作成した。

得られた薄膜EL素子の5KHz、正弦波駆動下のEL飽和輝度の相対値を第1表に示した。ここで輝度基準には比較例ZnS:0.01Tmを用いた。第1表から明らかなように、比較例では $b=0.01, 0.03, 0.06$ の順に輝度が大幅に減少するのに対して本発明のZn_{0.9}Mg_{0.1}S母体では、 $b=0.03$ すなわちTm濃度3モル%の時、ZnS:0.01Tmの209%の輝度が得られた。

ここで、上記組成物中に無視もしくは許容しうる範囲の他の不純物が存在することは本発明の要旨の範囲に含まれる。

例えば、Zn_{1-a}Mg_aS中にはMn等の不純物が固溶しやすい。また、蛍光体の焼成工程あるいは蒸着工程においてMnが上記組成に混入することがしばしば生ずる。しかし、このようなMnの混入は、少量に止まるかぎりにおいて、本発明の

蛍光体の特性劣化を惹起しない。また、無視し得ない量のMnが存在する場合においても、Mnの存在(例えば10モル%程度)に伴う発光が、EL素子等の白色表示に対して阻害要因とならない範囲においては、無視しうるものと同様に扱うことができる。

実施例3

EB蒸着法により、発光層が膜厚0.30 μ m、ピーク波長585nmのZnS:Mnと膜厚0.81 μ m、ピーク波長475nmの本発明のZn_{0.9}Mg_{0.1}S:0.33Tmの二層密着膜から成り、この発光層を実施例2と同じY₂O₃絶縁膜でサンドウィッチした薄膜EL素子を作成した。5KHz、正弦波駆動により、 $x=0.307, y=0.290$ 、色温度6500Kの昼光色発光を得た。また、上記二層密着層の膜厚をZnS:Mnでは0.45 μ m、本発明の蛍光体では0.62 μ mに選ぶことにより、 $x=0.410, y=0.365$ 、色温度3400Kの温白色発光を得た。得られた色度点の一例を第1図に黒点(符号4)で示す。同様に

な色度点は、上記二層密着膜によらずとも、ZnS:Mn蛍光体と本発明の蛍光体の同時EB蒸着によっても得られた。

第1図から明らかなように、黒点は色度図上でほぼ一直線上に並んでおり、本方法で得られた白色表示は、赤色、緑色、青色の三色混合から成る白色表示とは本質的に異なる安価な低次元白色であることを示している。

第1表

Tm濃度(モル)	MgS固溶量(モル)	
	n=0.0 [比較例]	a=0.1 [実施例2]
b=0.01	100	182
b=0.03	61.0	209
b=0.06	50.5	103

〔発明の効果〕

本発明によれば、低廉原料を用いて類似発光色の従来蛍光体よりも高輝度かつ高彩度の発光が得られるばかりでなく、通常構造の薄膜EL素子に適用して広域色温度の、しかも安価な白色表示が

得られるなどの効果があり液晶バックライトに好適である。

なお、本発明の効果は主発光がTm³⁺でもたらされる新規蛍光体組成物で得られるものであるが、Tm³⁺以外の希土類イオンが微量混入されていても発明の効果は損われることはない。

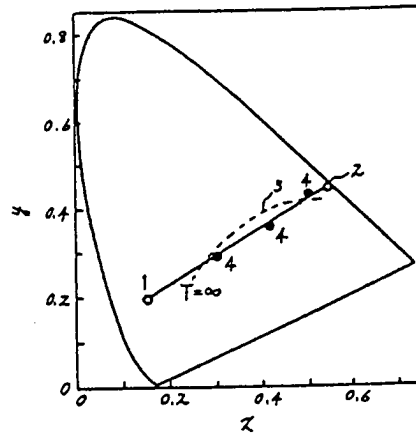
4. 図面の簡単な説明

第1図は本発明の実施例の蛍光体の色度特性を示す色度図である。

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图 1



- 1 本發明之螢光體之組成點
- 2 ZnS:Mn螢光體之組成點
- 3 點狀點
- 4 實施例3之EL素子之組成點

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	(22) Application Date	June 8, 1988
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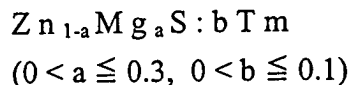
Details

1. Name of invention

Phosphor material and the manufacturing method of the phosphor material

2. Range of the patent claim

- 1) It is a phosphor material, which features that the main emission is generated by Tm^{3+} , which the ordinary formula is indicated below, and



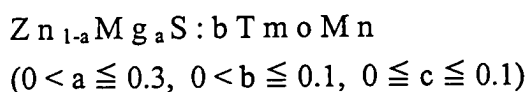
which the main composition is made of the solid solution related to zinc sulfate and magnesium sulfate.

- 2) It is concerning the phosphor material, which is stated in the aforementioned (1) of the range of the patent claim, and it is a phosphor material, which features that the numbers are indicated as below;

$$(0.05 < a \leq 0.2, 0 < b \leq 0.06)$$

- 3) It is a manufacturing method of phosphor material, which features that the main material is made of Tm, and the baking is performed with the designated amount of Zn and Mg raw material at 900 °C or higher.
- 4) It is an EL element, which features that at least one component of the emission layer, which composes the EL element, is made of the phosphor material, which is mentioned in (1) of the range of the patent claim.

- 5) It is an evaporation source target for the phosphor material film forming, which features that it shall obtain the phosphor material film, which shall include the composition of (1) of the range of the patent claim by forming each raw material of Zn, Mg and Tm, which are constructed by the sulfide.
- 6) It is an EL element, which features that the combination of the structure is made of the emission layer, which is mentioned in (4) of the range of the patent claim, and the orange emitting ZnS : Mn emission layer, which shall have the relationship of the complementary colors to the said emission layer.
- 7) It is a phosphor material, which features that the main emission is generated by Tm^{3+} , which the ordinary formula is indicated below, and



which the main composition is made of the solid solution related to zinc sulfate and magnesium sulfate.

3. Detailed explanation of the invention

[Utility field of the industry]

This invention is related to the phosphor material of the new composition, which the main emission is within the blue color area, and the manufacturing method of such phosphor material, and it is specially related to such phosphor material, which shows an excellent characteristic for using it in the blue color area of the white color EL display.

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[Existing technique]

There are publications according to the phosphor material, which shall have the (Zn, Mg) S as the main material and the examples are shown below. However, there are no publications concerning the phosphor material, which the Tm and Tm – Mn are added to the said main material.

- 1) The emitting characteristics of the ultraviolet light excitation and the electron beam excitation concerning the phosphor material, which Cu, Ag, Pb, As, Sb, Bi, Cu – Mn, Pb – Mn and Cu – Pb are added to the main material of (Zn, Mg) S, which is composed of MgS less than 25 mol% and ZnS, were written on page 155 to page 158, Vol. 99 (4) of the Journal of Electrochemical Society (1952).
- 2) The emitting at 450nm peak was observed by 290nm excitation by adding Mn of 50 ppm to the main material of $Zn_{1-a}Mg_aS$, $0 < a < 0.1$, which were mentioned on page 179 to page 183, Vol. 72 of the Journal of Crystal Growth (1985).
- 3) It is reported that the emission peak wavelength of the scattered EL, which shall use $Zn_{1-a}Mg_aS : Cu, Br$, shall be shifted from 525nm to 436 nm, on page 2100 to page 2108, Vol. 21 of the Journal of Material Science (1985).

- 4) Page 512 to page 516, Vol. 9 (4) of the Russian Journal of Inorganic Chemistry mentioned that the MgS shall solidly dissolve up to 22 mol% against the ZnS, and when the amount of the solid solution increases, a and C of the hexagonal grid invaluable shall also increase.
- 5) In the '80 S I D Digest, Page 106 (1980), concerning the thin film element, lm/W of $\text{ZnS} : \text{TmF}_3$ is 2 digits smaller than the lm/W of the $\text{ZnS} : \text{Mn}$.
- 6) In the KOKUKAISHO 61-49397, it indicates such full-color thin film EL element, which features that it consists of 2 substrates, which are the transparent substrates that one of the emitting EL elements of red, green or blue, is formed on, and another transparent substrate, which the emitting EL element of the remaining two colors are formed on, and that both substrates are arranged at the facing way to each other.

[The problem to be solved by the invention]

According to the abovementioned publications, the main emitting areas of the phosphor materials, which are mentioned in (1), (2) and (3) are all within the blue area, however, because the half value range of the emitting band is more than 70nm, the luminance is low as for the blue component of the color displays. Also, the thin film EL element, which is created using these phosphor materials, might not generate emission at all or even if they do, the luminance shall be extremely low.

On the other hand, although the luminance of the $\text{ZnS} : \text{TmF}_3$, which is mentioned in the above (5), is high, lm/W is more than 2 digits lower than $\text{ZnS} : \text{Mn}$, which is a weak point. Also, the abovementioned (6) has a weak point in that the element structure shall be complicated.

The purpose of this invention is to provide the new phosphor composition material, which shall solve all the problems mentioned above, and the manufacturing method of such phosphor material. The other purpose of this invention is to provide the EL element, which is possible to make the white displays. Also, one other purpose of this invention is to provide the evaporation source target composition material, which is suitable for forming the phosphor film of the abovementioned EL element phosphor material.

[The method of how to solve the problem]

The above purpose shall be accomplished by using the Tm^{3+} with high luminance against the emitting ion as well as expanding the ZnS related main grid in order to make it easy to introduce the Tm^{3+} .

Therefore, the composition of the phosphor of this invention is indicated by the ordinary formula of $\text{Zn}_{1-a}\text{Mg}_a\text{S} : b\text{Tm}$, and it shall obtain the most effective characteristics within the range of $0 < a < 0.3$, $0 < b \leq 0.1$.

In addition to the abovementioned composition, the fact that there are impurities, which are mixed during the manufacturing process, etc., they shall not cause deviation from the purpose of the invention.

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Also, by combining the phosphor film of the abovementioned composition and other phosphor film, an excellent white display EL element can be created.

As universally known, because the ion radius of Zn^{2+} is $r = 0.74 \text{ \AA}$ is smaller than the ion radius of Tm^{3+} of $r = 0.95 \text{ \AA}$, in order to introduce a designated amount of Tm^{3+} , it is necessary to expand the main grid. In order to expand the main grid, one should dissolve ZnS into MgS. For example, if 22 mol% MgS is dissolved into ZnS, the grid invaluability is increased by 1.9% and the grid invaluability Co is increased by 1.1%.

Also, in order to introduce the emitting ion of the triple positive charge such as Tm^{3+} to the positive ion grid point of the double positive charge such as Zn^{2+} and Mg^{2+} , instead of using Tm element, which is widely used, it is possible to use sulfuric raw material of Tm or cheap oxide raw material. By using the evaporation source target of the combination of the abovementioned sulfuric raw material, the white display EL element shall be created effectively.

[Function]

The most important advantage of this invention is that the same level or even higher luminance as the $ZnS : 0.001 Tm$, which the initial material is TmF_3 , is obtained by $Zn_{0.8}Mg_{0.1}S : 0.001 Tm$, which the initial material is Tm_2O_3 , which is a cheaper material.

Also, the second advantage is that by expanding the main grid from ZnS to $(Zn, Mg)S$, the adjustment of Tm density to make the suitable luminance becomes easier. This advantage is good for the adjustment of the Tm density within the emission layer of then thin film EL element, which makes the upper limit of the Tm density within the emission layer at least one digit higher than the existing number.

The third advantage is that by using sulfuric Tm instead of TmF_3 as the initial material, stable EB deposition can be performed without having any charge ups.

The abovementioned second and third advantages, which are related to the thin film EL element forming, shall generate the fourth advantage as well. It means by combining the blue emission layer with the optimum condition of the Tm density and well-known $ZnS : Mn$ orange emission layer, such white EL display as the color temperature of 3000K to 9000K can be possible. When the amount of MgS solid solution against ZnS is increased, a weak broad emitting appears in the orange color area besides the main emitting within the blue color area, and because this emitting shall be overlapped with the $ZnS : Mn$ orange color emitting, it shall not bother the white display.

[Example of implementation]

Hereinbelow, this invention shall be explained in accordance with the implementation example.

Implementation example 1

According to the ordinary formula of $\text{Zn}_{1-a}\text{Mg}_a\text{S} : b \text{ Tm}$, in order to compose the phosphor material of $a = 0.15$, $b = 0.001$ which means $\text{Zn}_{0.85}\text{Mg}_{0.15}\text{S} : 0.001 \text{ Tm}$, the initial material of ZnS, MgS and Tm_2O_3 with high purity shall be measured accurately, and shall be mixed them well, and after mixing, 100 g of them shall be placed on the transparent quartz board, and then they shall be baked at 1000 °C for 2 hours at the atmosphere of H_2S flow rate of 100 ml/min. Also, for the comparative example, Tm F_3 is used for the initial raw material, which is commonly used for $a = 0$, $b = 0.001$, which means $\text{ZnS} : 0.001 \text{ Tm}$, and baked under the exact same firing conditions as the above. The luminance by the electron beam excitation of 10 KV, $1 \mu\text{A} / \text{cm}^2$ was 119% setting the standard figure as the figure of the comparative example when using the phosphor material of $a = 0.15$. When $b = 0.001$, the luminance which is higher than the comparative example is obtained at $x = 0.05$, 1.0 and 2.0 of the chromaticity coordinates. On the other hand, when $a > 0.3$, because the association coloring appeared, which shall be the reason of the sludge of MgS, such luminance, which is equal to the comparative example, could not be obtained. Tm_2O_3 is a cheaper material than Tm F_3 , and it is a better material as for the utility phosphor material.

Implementation example 2

A 3-layer structured thin film EL element, which the emission layer is sandwiched between the Y_2O_3 insulation layers, is created by the EB deposition technique. The phosphor material of this invention and the $\text{ZnS} : \text{Tm}$ phosphor material of the comparative example are used for the emission layers. According to the phosphor composing of this invention, sulfuric Tm, which the sulfate rate is 93% from sulfurating Tm_2O_3 for 45 hours at the atmosphere of H_2S 100 ml / min., is used for the initial material of Tm in order to control the charge up when performing the EB deposition. Therefore, unlike the implementation example 1, all of the Zn, Mg and Tm raw materials are sulfuric, and the evaporation source target for EB was created by firing at 1000 °C at the Algon atmosphere after applying the pressure molding on the powder phosphor material, which is obtained by baking the mixture of each designated amount of Zn, Mg and Tm material for 2 hours at 900 to 1000 °C at the H_2S flow rate of 100 ml / min.

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On the other hand, the initial material of Tm concerning the comparative example of $\text{ZnS} : \text{Tm}$ is Tm F_3 , which is the same as the implementation example 1, and the evaporation source target is created by the same process as the abovementioned pressured molding baking.

The relative value of the EL saturated luminance under the sine wave drive at 5 KHz of the obtained thin film EL element is shown in Table 1. The comparative example of $\text{ZnS} : 0.01 \text{ Tm}$ is used for the luminance standard. As it is clearly seen from Table 1,

compared with the result of the comparative example, which the luminance decreases from $b = 0.01$ to 0.03 and then to 0.06 when $b = 0.03$, which means Tm density is 3 mol%, 209% luminance of ZnS : 0.01Tm is obtained from the Zn 0.9 Mg 0.1 S main material of this invention.

The existence of the impurities within the abovementioned composition, which can be ignored or within the acceptable range, shall be included in the range of this invention. For example, the impurities of Mn, etc., are easily dissolved in the $\text{Zn}_{1-a}\text{Mg}_a\text{S}$. Also, Mn often shall be mixed with the abovementioned composition during the firing process of the phosphor material or deposition process. However, such mixture of the Mn shall not bring the quality inferior to the phosphor material as long as it stays within the certain amount, and if the emitting from the existence of Mn (for example 10 mol%) shall not be the reason for an impediment against the white color display of the EL element, etc., it can be treated as the same case, which can be ignored.

Implementation example 3

The thin film EL element, which the emission layer is sandwiched by the same insulation film Y_2O_3 as the implementation example 2, which is made of the 2-layer density film, which is composed of the ZnS : Mn, which the film thickness of the emission layer is $0.30\text{ }\mu\text{m}$ and the peak wavelength is 585 nm, and the Zn 0.8 Mg 0.1 S : 0.33 Tm of this invention, which has the film thickness of $0.81\text{ }\mu\text{m}$ and the peak wavelength of 475 nm, is created by the EB deposition technique. By the sine wave drive of 5KHz, the day light color emitting of the color temperature of 6500K, $x = 0.307$ and $y = 0.290$ were obtained. Also, by selecting the film thickness of the abovementioned 2-layer density layer to be $0.45\text{ }\mu\text{m}$, in the case of ZnS : Mn, and $0.62\text{ }\mu\text{m}$, in the case of the phosphor material of the invention, $x = 0.410$, $y = 0.365$, and the color temperature of 3400K, warm white color emitting was obtained. One example of the obtained chromaticity point is shown as the black points (symbol 4) in Figure 1. The similar chromaticity point is obtained also by the simultaneous implementation of EB deposition technique to the ZnS : Mn phosphor material and the phosphor material of this invention even if it does not depend on the abovementioned 2-layer density film.

As it is clearly seen from Figure 1, the black points are arranged almost in a straight line, and the white display, which is obtained by this method shows that it is a low dimension white color, which is low cost and essentially different from the white display, which is composed of the mixture of the red color, green color and blue color.

Table 1 (P.632)

M g S solid Solution Amount (mol)	a = 0.0	a = 0.1
Tm density (mol)	[Comparative example]	[Implementation example 2]
b = 0.01	100	182
b = 0.03	61.0	209
b = 0.06	50.5	103

[Effectiveness of the invention]

According to the invention, not only does this obtain higher luminance and higher tint, but also it obtains wide color temperature, which is suitable for the thin film EL element of the ordinary structure, as well as the low cost white display, which is excellent for the liquid back light.

Further, the effectiveness of the invention is obtained by the new phosphor material composition, which is made of Tm^{3+} , however, the effectiveness of the invention shall not change even if a small amount of ion besides Tm^{3+} is mixed.

4. Simple explanation of the figure

Figure 1 is a chromaticity diagram, which indicates the chromaticity characteristics of the phosphor material of the implementation examples of the invention.

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Figure 1

- 1: Chromaticity point of the phosphor material of the invention
- 2: Chromaticity point of the ZnS : Mn phosphor material
- 3: Locus of the black points
- 4: Chromaticity point of the EL element of the implementation example 3

End